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Development of a Non-radioactive
Tracer for Use in Bioaquatic Environments

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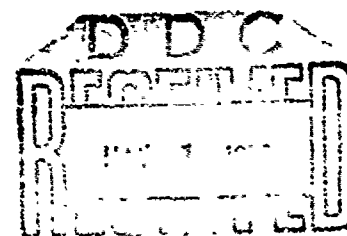
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13. ABSTRACT <p>The addition of a non-radioactive material to a river as a tracer has been considered. The basic problem is to recover a reasonable size water sample (in the order of a gallon) downstream from the point of addition and specifically identify the added tracer. A variety of parameters are involved, but with the high sensitivity of analytical techniques available today, many different tracer materials should be practical. One possible method suggest here includes utilizing a gas chromatograph and a sensitive mass spectrometer for positive identification of the tracer.</p> <p>A specific substance, benzyl chloride, was added to the river where calculations including dilution volume, flow rate and recovery factors were estimated and recovery of some of the tracer was anticipated. Identification of the tracer, however, was inconclusive in four trial runs.</p> <p>As a practical tracer technique, the method seems very promising, even with large dilutions, for certain types of compounds. A lack of information on river parameters probably prevented proper pick-up and identification in the few runs made.</p>			

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Introduction

The objective in this research effort was to determine the feasibility of addition of a stable chemical compound into a large volume of water such as a river, with subsequent recovery of a sample of the water and positively identifying the original added compound as a trace impurity in the water.

This objective which is in essence a non-radioactive tracer technique, could have many applications which are not elaborated on here. For example, the flow characteristics of a stream could be measured. The chemical interaction of certain compounds could be determined with regard to the river water and its natural impurities.

In this particular study a few grams of tracer material labelled with a non-radioactive isotope would be a reasonable quantity; recovery would be needed of a sample of possibly a gallon or so (about 4 liters). However, since the material is not to be a radioactive tracer, the alternative for the positive identification is the non-radioactive labelled compound, which requires the identification ultimately by use of mass spectrometry. In addition the compound can not be one which is likely to be found in very large quantities in a river.

In approaching such a problem a variety of alternatives were considered from types of compounds, to recovery and enrichment techniques, and, of course, method of detection. Certain criteria

had to be established arbitrarily, or by judicious choice, or by the limitations of the available equipment, time and money.

Discussion and Results

An outline of the steps taken and reasons are included here. The individual experimental techniques are not particularly unique and are given in detail.

The selection of a compound to be used as a tracer was a particularly difficult problem because of the many variables to be considered. The compound to be used had to be labelled either with carbon-13 or deuterium for practical reasons. Since carbon 13 is abundant in nature to the extent of 1% of carbon 12, all single carbon atom molecules contain 1% labelled carbon 13 such as natural methane, hydrogen cyanide or formaldehyde. Molecules containing two carbon atoms, contain 2% C-13 labelled molecules naturally, but only 0.01% where both carbon molecules are C-13. For other fully labelled compounds, that is where all carbon atoms are C-13, the natural abundance can be calculated by statistics as roughly 10^{-6} fraction for three carbon atom compounds (e.g. propane), 10^{-8} for four (e.g. Butane), and 10^{-10} for five (e.g. pentane).

It was thought most reasonable to use any fully C-13 labelled component with four or more carbons, since it could be positively identified as not originating from a natural compound even when diluted by a million to one.

The companies offering such labelled compounds are limited, however, and delivery was considered questionable. As an

alternative a deuterated compound was considered and final chosen where a large number are relatively readily available. Delivery was, even then, several months delayed.

Benzyl chloride fully deuterated was finally chosen because:

1. It appeared to be available in a reasonable time
2. Sufficiently soluble in water
3. Sufficiently stable against exchange with hydrogen (This is no problem with C-13 labelling and therefore a C-13 labelled compound would be preferred)
4. Reasonably low vapor pressure
5. Slightly heavier than water
(4 and 5 are important since many compounds may simply spread out on a water surface and evaporate)
6. Detectible by gas chromatography in concentrations of less than about 30 parts per billion in a microgram of benzene.

Further benzyl chloride could be conveniently extracted with benzene from river samples and enriched by distillation in a micro-distillation column using very fine Fenske packing. The heavier fraction enriched in benzyl chloride could then be analyzed utilizing the gas chromatograph and the benzyl chloride fraction recovered at the end on the column and analyzed mass spectrometrically.

It was found that many organic peaks are observed in the chromatogram from the extracted fraction and it was gratifying that

no major peak was observed in the region or vicinity of the benzyl chloride peak which could have severely limited the detection capability.

In principle the overall problem is relatively simple; that is, add the tracer compound, recover a diluted portion, extract it, and measure it with a highly sensitive analytical technique. However, each step in progress required considerable study and evaluation. All of the refinements now used were not developed until the last field test was made. It was felt that the natural variations occurring in the river were probably the largest single factor in preventing anything, but marginal and inconclusive observations. The expensive labelled benzyl chloride was not used, however, since the preliminary field tests were unsuccessful.

Conclusions

It is considered that all laboratory techniques needed here for employment of a single tracer (deuterated benzyl chloride), have been perfected for application to field testing. The first trials in the field were made without all of the refinements which have now been worked out. The technique now also permits a much larger number of samples to be handled. In a fast flowing river stream, it is necessary to obtain a larger number of samples for recovery and analysis.

If further testing is desired a period covering from spring through late fall, six to eight months should give more than sufficient time to obtain positive field results. In principle no more than a week is needed, but the initial field tests prove the need for a more cautious and conservative approach.